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THERMALLY-COUPLED COLUMNS: DISTILLATION

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Introduction

A considerable amount of energy is used in distillation operations. Energy integration has proven to be successful in reducing energy costs for conventional distillation arrangements. However, the scope for energy integration of conventional distillation columns into an overall process is often limited. Also, practical constraints often prevent integration of distillation columns with the rest of the process.

If the column cannot be integrated with the rest of the process or, if the potential for heat integration is limited by the heat flows in the background process, then we must turn our attention back to the distillation operation itself and look at unconventional arrangements.

Figure 1 shows two conventional arrangements for the separation of a three-component mixture. The sequence shown in Figure 1A is the so-called direct sequence, in which the lightest component is taken overhead in each column. The indirect sequence shown in Figure 1B takes the heaviest component as bottom product in each column.

One of the most significant unconventional arrangements involves thermal coupling. **Figure 2** shows a number of unconventional arrangements that use thermal coupling. In thermal coupling part of the heat transfer necessary for the separation is provided by direct contact via material flows. Figure 2A shows a side-rectifier arrangement and Figure 2B a side-stripper arrangement. Arrangements similar to that in Figure 2B are widely used in petroleum refining. The fully thermally coupled arrangement in Figure 2C (sometimes known as the Petlyuk column) has been known for over 50 years. Various studies have shown that thermally coupled arrangements can save up to 30% of energy costs when compared with conventional arrangements.

Simple Versus Complex Columns

Consider first the design of distillation systems comprising only simple columns. These simple columns employ:

- one feed split into two products;
- key components which are adjacent in volatility;
- a reboiler and a condenser.

For a three-component mixture in which simple columns are employed, the decision is between the two sequences illustrated in Figure 1.

Consider the first characteristic of simple columns, which involves a single feed being split into two products. As a first option to two simple columns, the possibilities shown in **Figure 3** can be considered, in which three products are taken from one column. The designs can be both feasible and cost-effective when compared with simple arrangements, but only for certain conditions. If the feed is dominated by the middle product (typically more than 50% of the feed) and the heaviest product is present in small quantities (typically less than 5%) then the arrangement shown in Figure 3A can be an attractive option. If a pure middle product is required, then it is usually only possible if there is a large volatility difference between components B and C, with the middle product taken as a vapour to assist the separation. The heavy product must find its way down the column past the side-stream. Unless the heavy product has a small flow and the middle product a high flow, a reasonably pure middle product cannot be achieved.

If the feed is dominated by the middle product (typically more than 50%) and the lightest product is present in small quantities (typically less than 5%), then the arrangement shown in Figure 3B can be an attractive option. This time the light product must find its way up the column past the side-stream. If

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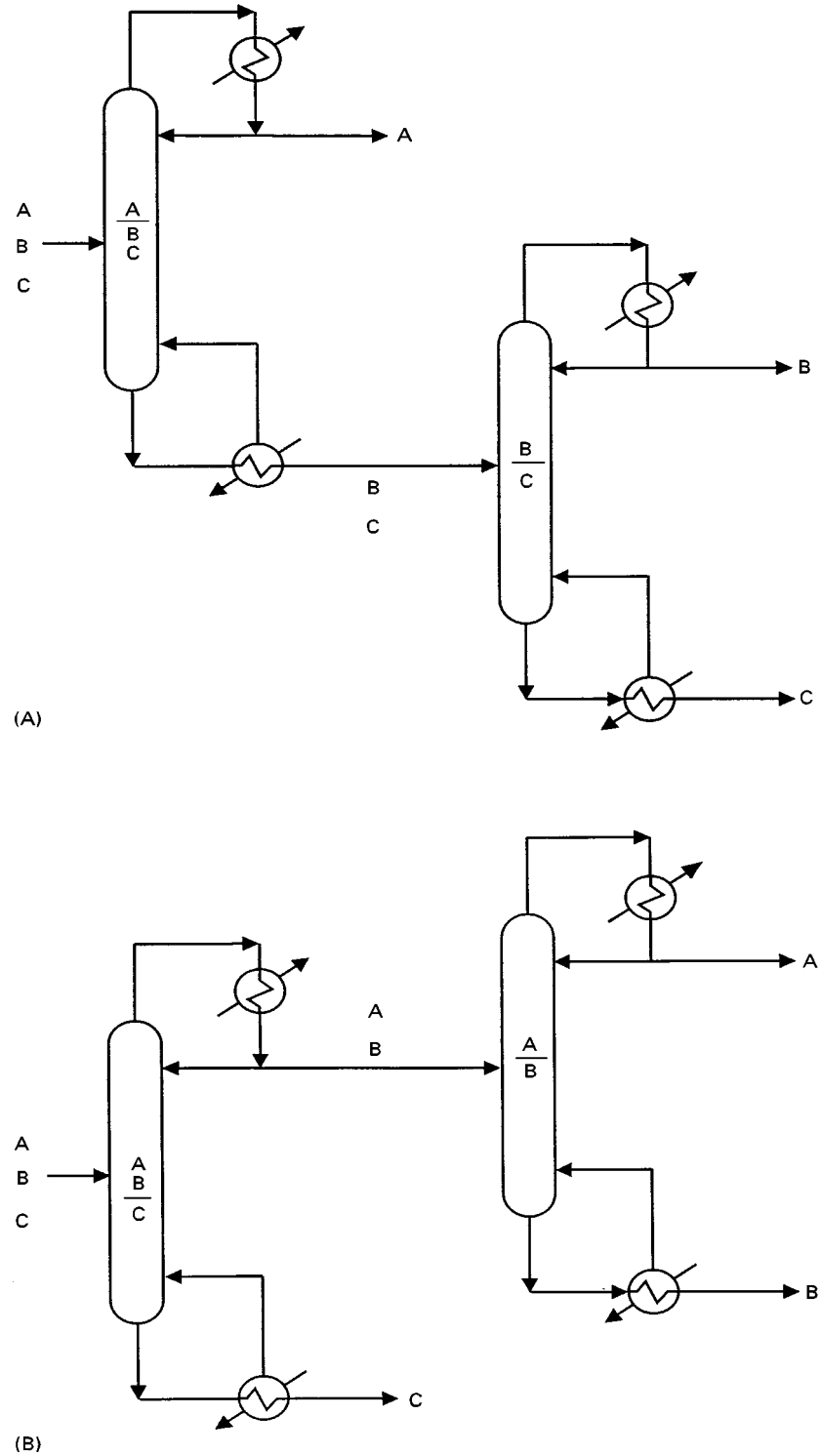

Figure 1 The (A) direct and (B) indirect sequences of simple distillation columns for a three-component separation. (Reproduced with permission from Triantafyllou and Smith (1992) *Transactions of the Institution of Chemical Engineers, Part A* 70: 1992.)

a pure middle product is required, then it is usually only possible if there is a large volatility difference between components A and B, with the middle product taken as a liquid to assist the separation.

In summary, single-column side-stream arrangements can be attractive when the middle product is in excess and one of the other components is present in only minor quantities. Thus, the side-stream column

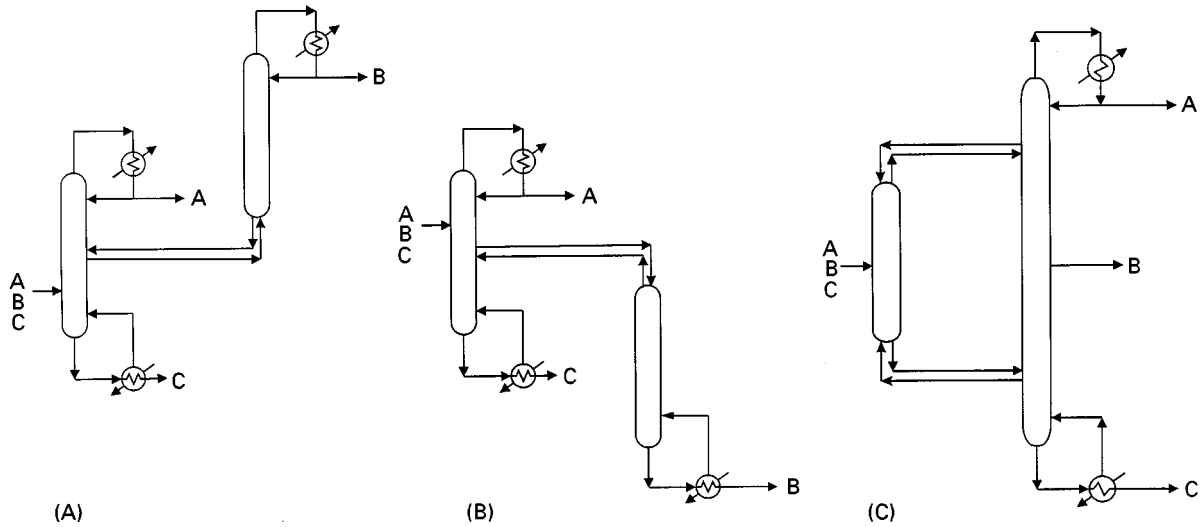

Figure 2 Thermally coupled columns. (A) Side-rectifier; (B) side-stripper; (C) fully thermally coupled column. (Reproduced with permission from Triantafyllou and Smith (1992) *Transactions of the Institution of Chemical Engineers, Part A* 70, 118.)

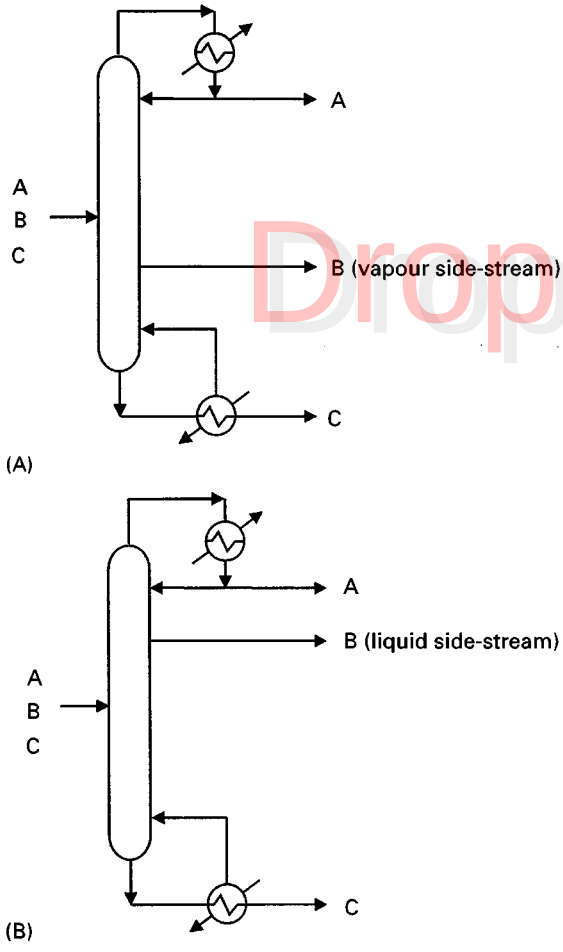

Figure 3 Distillation columns with three products. (A) More than 50% middle component and less than 5% heaviest component; (B) more than 50% middle component and less than 5% lightest component. (From Smith and Linnhoff (1988) *Chemical Engineering Research and Design*, 66, 195, reproduced with permission from the Institution of Chemical Engineers.)

only applies to special feed compositions. More generally applicable arrangements are possible by relaxing the restriction that separations must be between adjacent key components.

Consider a three-product separation as shown in **Figure 4A** in which the lightest and heaviest components are chosen to be the key separation in the first column. In such a case, two further columns are required to produce pure products. However, note that the bottoms and overheads of the second and third columns in Figure 4A are both pure B. Hence the second and third columns could simply be connected and product B taken as a side-stream, as shown in Figure 4B. The arrangement in Figure 4B is known as a prefractionator arrangement. Note that the first column in Figure 4B, the prefractionator, has a partial condenser to reduce the overall energy consumption. The prefractionator arrangement in Figure 4B typically requires 30% less energy than conventional arrangements for the same separation duty. The extent of the energy saving depends on the feed composition and the relative volatility of the components being separated. The energy saving results from the fact that the prefractionator arrangement is thermodynamically more efficient than a simple arrangement.

To understand why this is the case, consider the sequence of simple columns shown in **Figure 5**. In the direct sequence shown in Figure 5, the composition of component B in the first column increases below the feed as the more volatile component A decreases. However, moving further down the column, the composition of B decreases again as the composition of the less volatile component C increases. Thus, the composition of B reaches a peak, only to be remixed.

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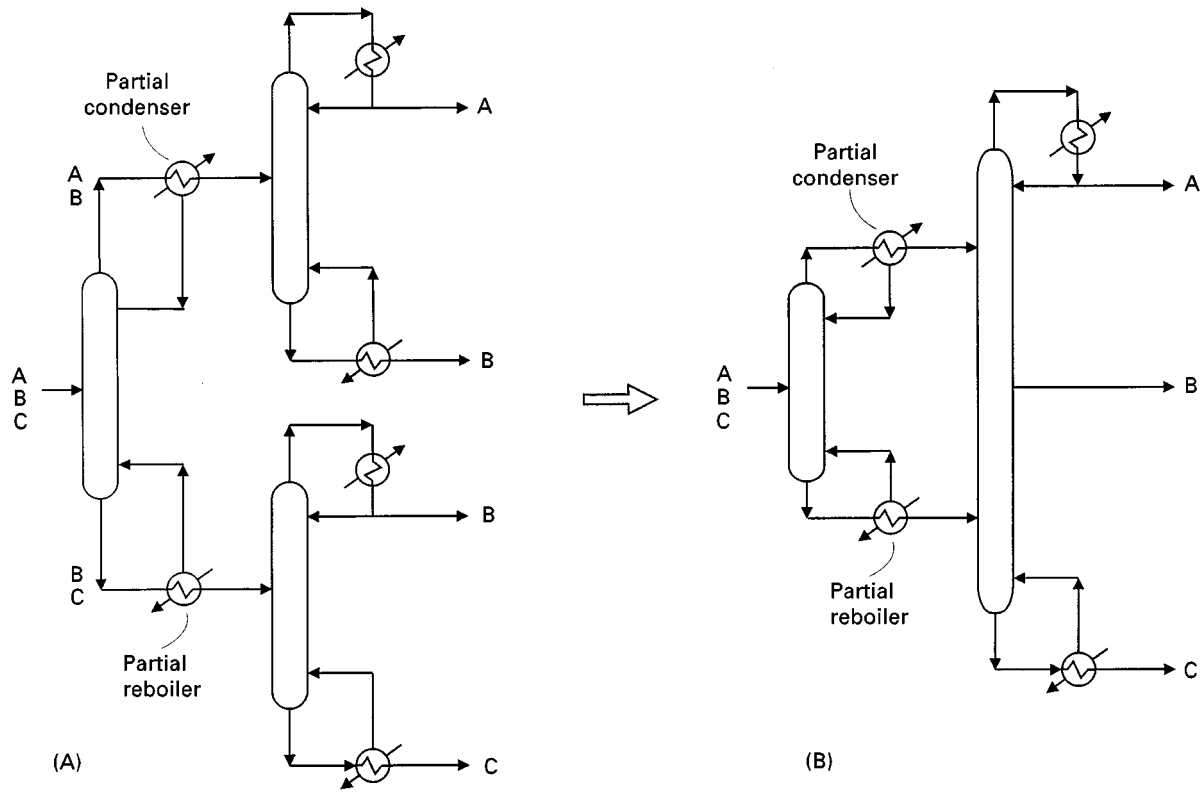

Figure 4 Choosing nonadjacent keys leads to the prefractionator arrangement. (A) Sequence for three product separation using nonadjacent keys; (B) prefractionator arrangement. (Reproduced with permission from Smith (1995) *Chemical Process Design*, McGraw-Hill.)

Similarly, with the first column in the indirect sequence, the composition of B first increases above the feed and reaches a maximum only to decrease as the more volatile component A increases. Again, the composition of B reaches a peak, only to be remixed.

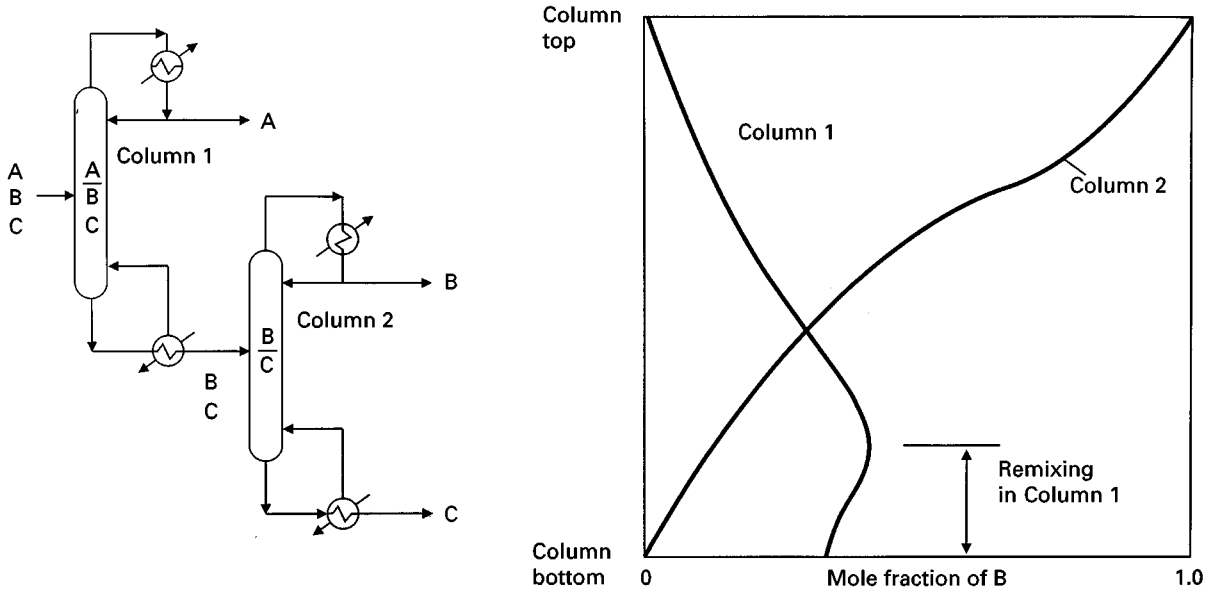

Figure 5 Composition profiles for the middle product in the columns of the direct sequence show remixing effects. (From Triantafyllou and Smith (1992) *Transactions of the Institution of Chemical Engineers, Part A* 70, 118, reproduced by permission of the Institution of Chemical Engineers.)

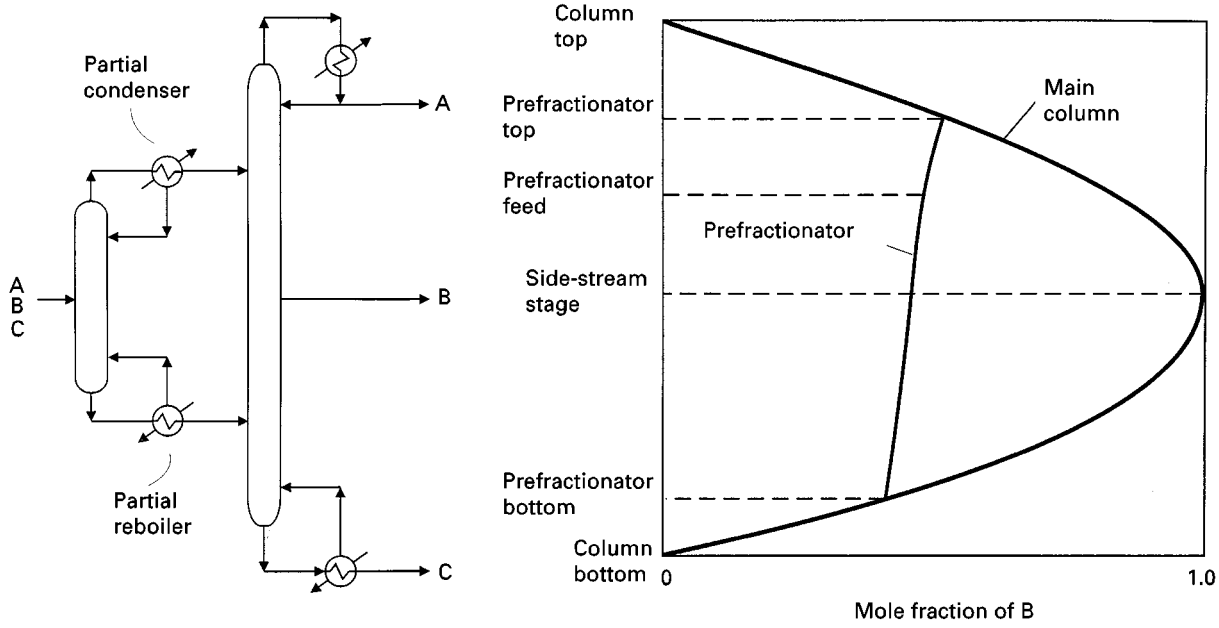

Figure 6 Composition profiles for the middle product in the prefractionator arrangement show that there are no remixing effects. (From Triantafyllou and Smith (1992) *Transactions of the Institution of Chemical Engineers, Part A* 70, 118, reproduced by permission of the Institution of Chemical Engineers.)

This remixing which occurs in both sequences of simple distillation columns is a source of inefficiency in the separation. By contrast, consider the prefractionator arrangement shown in **Figure 6**. In the prefractionator, a crude split is performed so that component B is distributed between the top and bottom of the column. The upper section of the prefractionator separates AB from C, whilst the lower section separates BC from A. Thus, both sections remove only one component from the product of that column section and this is also true for all sections of the main column. In this way, the remixing effects which are a feature of both simple column sequences are avoided.

In addition, one other feature of the prefractionator arrangement is important in reducing mixing effects. Losses occur in distillation operations due to mismatches between the composition of the column feed and the composition on the feed tray. Because the prefractionator distributes B between top and bottom, this allows greater freedom to match the feed composition with one of the trays in the column to reduce mixing losses at the feed tray.

Distillation Using Thermal Coupling

Rather than each column having a reboiler and condenser, it is possible to use material flows to provide some of the necessary heat transfer by direct-contact thermal coupling.

First consider thermal coupling of the simple sequences from Figure 1. **Figure 7A** shows a thermally coupled direct sequence in which the reboiler of the first column is replaced by thermal coupling. Liquid from the bottom of the first column is transferred to the second as before, but now the reboiler of the second column supplies the vapour required by the first column. The four column sections marked as 1, 2, 3 and 4 in Figure 7A can be rearranged to form a side-rectifier arrangement, as shown in Figure 7B.

Similarly, **Figure 8A** shows a thermally coupled indirect sequence in which the condenser of the first column is replaced by thermal coupling. The four column sections marked as 1, 2, 3 and 4 in Figure 8A can again be rearranged, but this time forming a side-stripper arrangement.

Both the side-rectifier and side-stripper arrangements have been shown to reduce the energy consumption compared with simple two-column arrangements. This results from reduced mixing losses in the first (main) column. As with the first column of the simple sequence, a peak in composition occurs with the middle product, but now advantage of the peak is taken by transferring material to the side-rectifier or side-stripper.

Side-stripper arrangements are commonly used in petroleum refinery separations. **Figure 9A** shows a typical arrangement for distillation of crude oil. The main column is fed with the pre-heated crude oil feed. Products are taken from various points from the main

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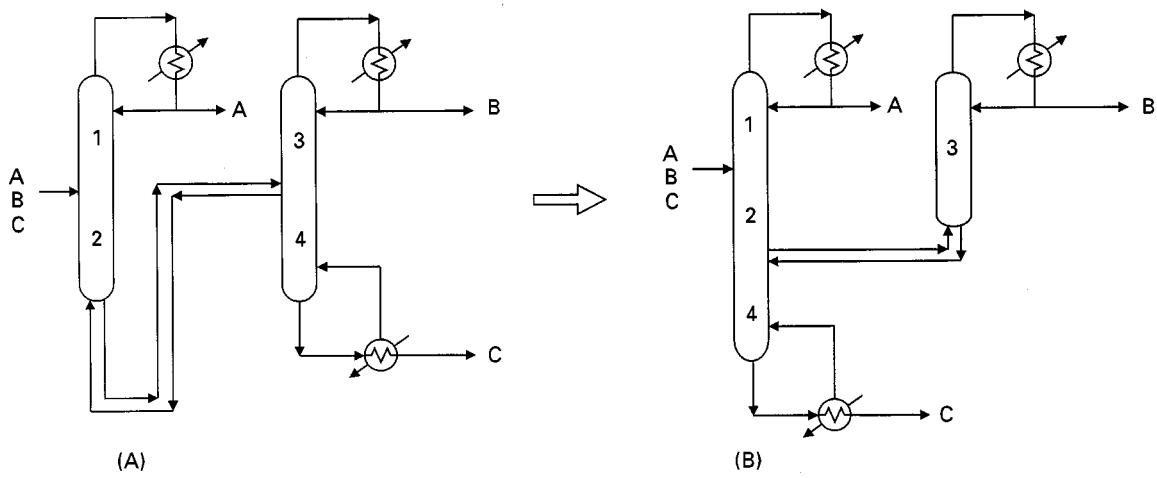

Figure 7 Thermal coupling of the direct sequence. (A) Thermally coupled direct sequence; (B) side-rectifier arrangement. (Reproduced with permission from Smith (1995) *Chemical Process Design*, McGraw-Hill.)

column via side-stripper columns. Heat is also removed at various points through the main column via pumparounds. Pumparounds take liquid from the column, cool it and return it to the column at a higher point, effectively acting as intermediate condensers. Heat to the side-strippers is supplied from either reboilers or live steam injection. The arrangement shown in Figure 9A is equivalent to a sequence of simple columns in the indirect sequence, as shown in Figure 9B.

Consider now thermal coupling of the prefractionator arrangement from **Figure 10A**. Figure 10B shows the equivalent thermally coupled prefractionator arrangement, sometimes known as the Petlyuk column. To make the two arrangements in Figure 10 equivalent, the thermally coupled prefractionator requires extra plates to substitute for the prefractionator condenser and reboiler.

Various studies have shown that the thermally coupled arrangement in Figure 10B requires typically 30% less energy than a conventional arrangement using simple columns. The saving depends on the feed mixture. In most cases the fully thermally coupled column in Figure 10B requires less energy than the side-rectifier and side-stripper arrangements, for the same separation. The prefractionator arrangement in Figure 10A and the thermally coupled prefractionator (Petlyuk column) in Figure 10B are similar in terms of total heating and cooling duties, but there are differences in the temperatures at which the heat is supplied and rejected.

Figure 11 shows the evolution from the prefractionator in Figure 11A to the thermally coupled prefractionator in Figure 11B. Finally, in Figure 11C, the thermally coupled prefractionator uses a single shell with a vertical baffle dividing the central section

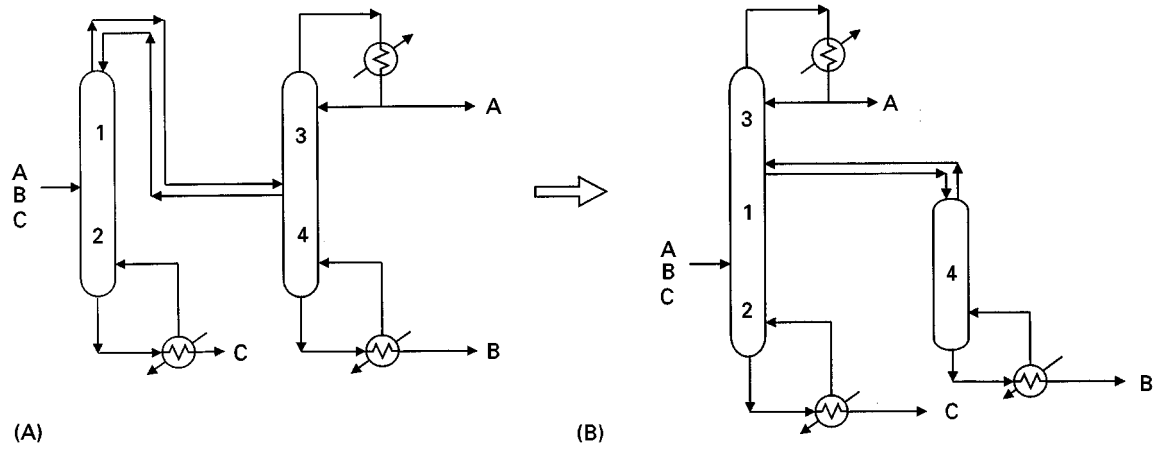

Figure 8 Thermal coupling of the indirect sequence. (A) Thermally coupled indirect sequence; (B) side-stripper arrangement. (Reproduced with permission from Smith (1995) *Chemical Process Design*, McGraw-Hill.)

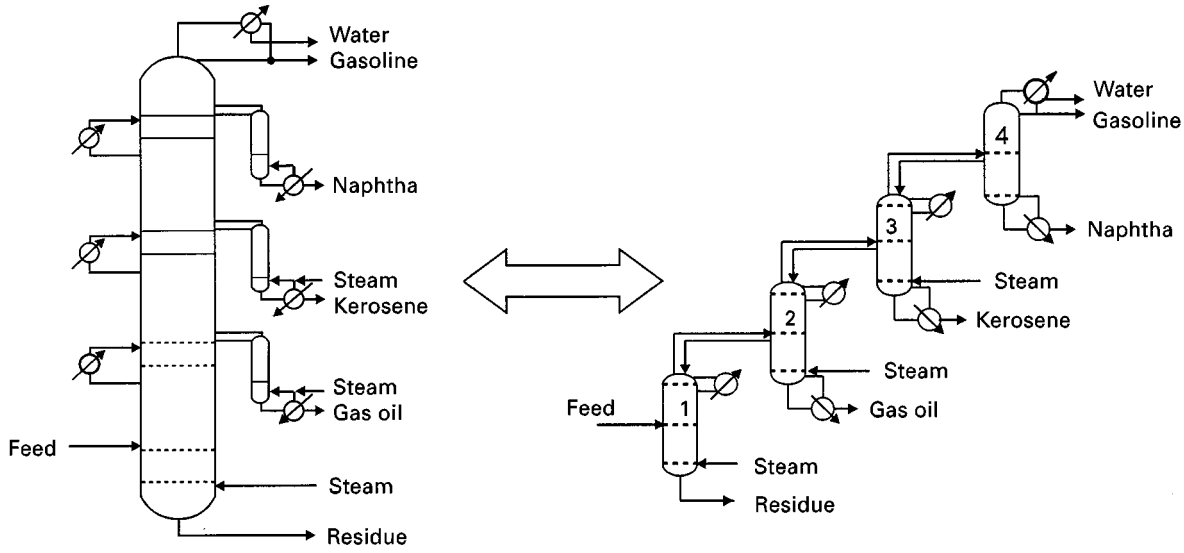

Figure 9 The typical crude oil distillation column decomposes to a sequence of simple columns in the indirect sequence.

of the shell into two parts, known as the dividing wall column or partition column. The arrangements in Figure 11 require almost the same energy consumption, which typically is 30% less than a conventional arrangement. However, in the case of the prefractionator in Figure 11A, the heat load is supplied at two points and rejected from two points. In addition, the dividing wall column in Figure 11C requires typically 30% less capital cost than a two-column arrangement of simple columns.

Dividing Wall Columns

Dividing wall columns, as shown in Figure 11C, have been known for over 50 years and yet it is only recently that they have been applied in practice. It is

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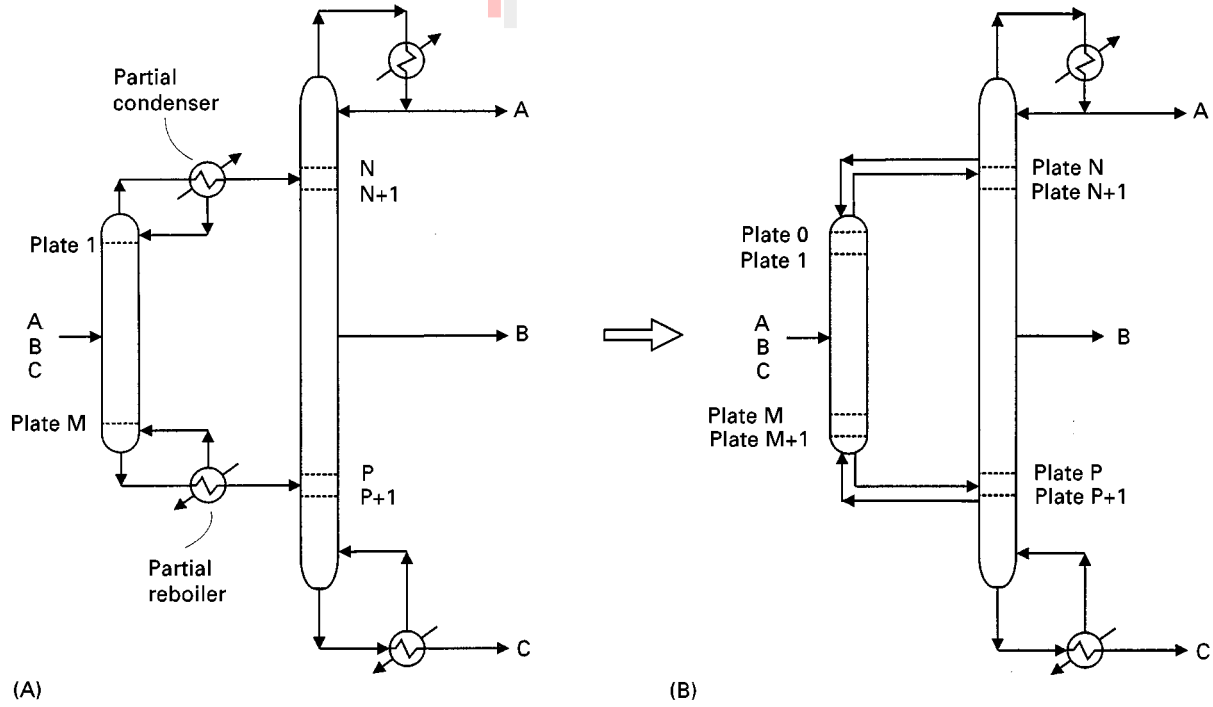

Figure 10 Thermal coupling of the prefractionator arrangement. (A) Prefractionator; (B) thermally coupled prefractionator. (Reproduced with permission from Smith (1995) *Chemical Process Design*, McGraw-Hill.)

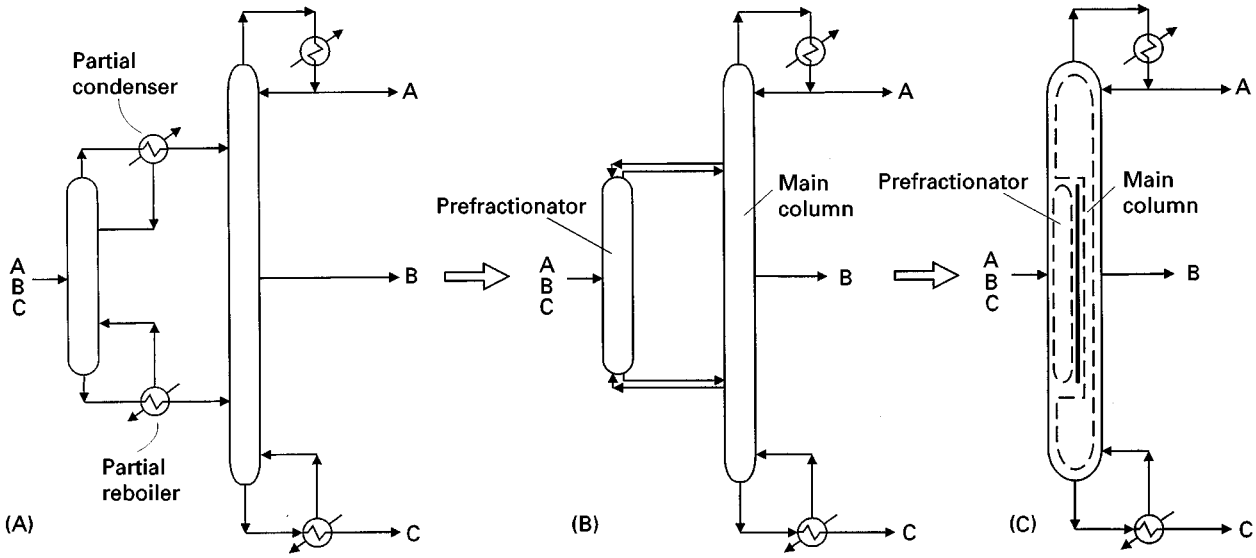

Figure 11 The thermally coupled prefractionator can be arranged in a single shell. (A) Prefractionator arrangement; (B) thermally coupled prefractionator (Petlyuk column); (C) dividing wall column. (Reproduced with permission from Smith (1995) *Chemical Process Design*, McGraw-Hill.)

true that the basic design is more problematic than a single conventional column, because there are more degrees of freedom in the design. However, methods have been developed to initialize the degrees of freedom prior to detailed simulation. Detailed simulation of the dividing wall column is carried out by modelling it as a Petlyuk arrangement, as shown in Figure 10B. Control of the column has also been a concern. However, such concern is misplaced, as the control is straightforward, being effectively the same as control of a side-stream column. Standard temperature and composition control configuration schemes can be employed. The hardware and column internals for the dividing wall column are also standard, despite the presence of the dividing wall. However, it should be noted that the column performance

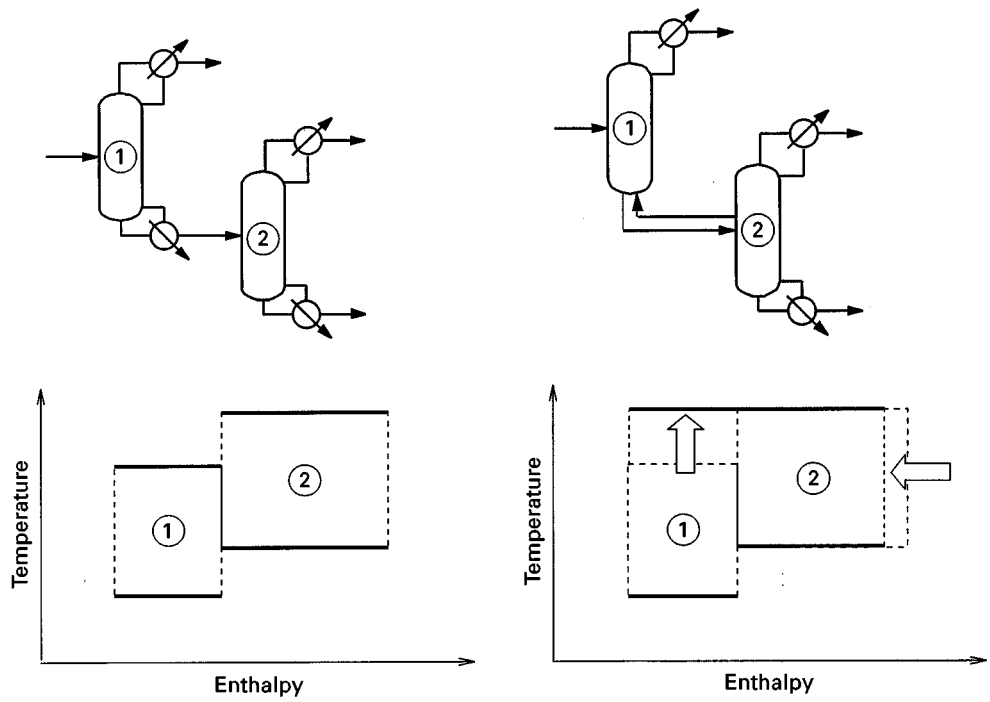

Figure 12 Thermal coupling reduces the quantity of energy required but makes temperatures more extreme.

suffers if the dividing wall is not insulated in some way. This can be done in practice by using two plates separated by a layer of insulation.

Temperature of Heat Supply and Rejection

So far the benefits of thermal coupling have been discussed in terms of the reduced energy required. Let us now consider the temperature at which the heat needs to be supplied and rejected if thermal coupling is used. It is always preferable to add the heat to the reboiler at the lowest temperature possible and to reject heat from the condenser at the highest temperature possible. In the first instance, this allows cheaper hot and cold utilities. In addition, if heat integration of the reboiler and condenser is to be considered, heat integration will also always benefit from lower reboiler temperatures and higher condenser temperatures.

Figure 12 compares a conventional and a thermally coupled arrangement in terms of temperature and enthalpy. In the conventional arrangement there is freedom to choose the pressures of the two columns independently, and thus the temperatures of the two condensers or the two reboilers can be varied independently. In the case of the thermally coupled arrangement no such freedom exists. Although the thermally coupled arrangement requires a smaller heat load than the conventional arrangement, more of the duties are at extreme levels. The smaller duties work to the benefit of utility costs and heat integration but the more extreme levels work against them.

Summary

Thermally coupled distillation columns offer considerable benefits in terms of operating costs. Side-stripper, side-rectifier and fully thermally coupled arrangements such as the Petlyuk column can save typically 30% of the energy consumption compared with sequences of simple columns. The magnitude of the saving depends on the feed composition and relative volatility of the components being separated. The dividing wall column also offers large potential savings in capital cost. Apart from the use of side-stripper arrangements in the petroleum refinery industry there has been reluctance on the part of process designers to exploit the full potential of thermal coupling. Control of thermally coupled arrangements does not present any particularly difficult problems.

See also: **II / Distillation:** Energy Management; Modelling and Simulation; Theory of Distillation.

Further Reading

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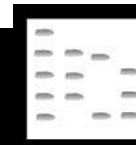
THIN-LAYER CHROMATOGRAPHY – VIBRATION SPECTROSCOPY



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Introduction

The utility of vibrational spectroscopy in chemical structure elucidation of separated thin layer chromatography (TLC) spots has been recognized for many years. Although the traditional method has been infrared spectroscopy (Fourier transform infrared spectrometry (FTIR)) a number of competing techniques now exist including normal Raman scattering (RS), Raman microspectroscopy (Micro-Raman), Fourier transform Raman (FT-Raman) and surface-enhanced Raman scattering (SERS). Since each type of spectra provide essential vibrational profile of analytes from the TLC plate, the different disciplines are natural partners in a general spectroscopic analysis. All methods involve the vibrational energy of the molecule and thus provide molecular and structural information about the separated sample. However, since infrared (IR) absorption, Raman scattering and SERS have different selection rules – what is frequently strong in